
REFERENCE

Morris,R.G.; Sallustio,B.C.; Saccoia,N.C.; Mangas,S.; Fergusson,L.K.; Kassapidis,C. Application of an improved HPLC perhexiline assay to human plasma specimens, *J.Liq.Chromatogr.*, **1992**, *15*, 3219–3232.

SAMPLE

Matrix: blood

Sample preparation: 400 μ L Plasma + 400 μ L 1 M diammonium hydrogen phosphate + 50 μ L 2.5 μ g/mL di-n-hexylamine in water + 100 μ L 1 mg/mL diisopropylethylamine + 5 mL isopentane:dichloromethane 60:40, vortex for 1.5 min, centrifuge at 1500 g for 5 min, freeze in dry ice/acetone. Remove the organic layer and evaporate it to dryness under reduced pressure at room temperature, reconstitute the residue in 100 μ L 10 mg/mL trans-4-nitrocinnamoyl chloride in dry MeCN, vortex for 15 s, let stand at room temperature for 30 min, add 100 μ L 25 mM sodium carbonate, vortex for 15 s, let stand at room temperature for 5 min, add 100 μ L MeCN:50 mM ammonium acetate 50:50, vortex for 30 s, centrifuge at 1500 g for 5 min, inject a 130 μ L aliquot of the supernatant.

HPLC VARIABLES

Column: 250 \times 4.6 5 μ m Spherisorb phenyl

Mobile phase: MeCN:water:glacial acetic acid 46:53.5:0.5

Flow rate: 2

Injection volume: 130

Detector: UV 340

CHROMATOGRAM

Retention time: 18.4

Internal standard: di-n-hexylamine (10.2)

Limit of detection: 30 ng/mL

OTHER SUBSTANCES

Extracted: metabolites

Simultaneous: dealkylidisopyramide, desethylamiodarone, desipramine, flecainide, mexiletine, nortriptyline, protriptyline, sotalol

KEY WORDS

derivatization; plasma

REFERENCE

Grgurinovich,N. Method for the analysis of perhexiline and its hydroxy metabolite in plasma using high-performance liquid chromatography with precolumn derivatization, *J.Chromatogr.B*, **1997**, *696*, 75–80.

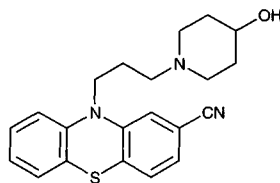
Pericyazine

Molecular formula: C₂₁H₂₃N₃OS

Molecular weight: 368.47

CAS Registry No.: 2622-26-6

Merck Index: 7306



SAMPLE

Matrix: solutions

Sample preparation: Prepare a 10 μ g/mL solution in MeOH, inject a 20 μ L aliquot.

HPLC VARIABLES

Column: 125 \times 4.9 Spherisorb S5W silica

Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

Flow rate: 2

Injection volume: 20

Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM

Retention time: 2.1

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazepine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipranone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserin, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamlamine, mecllophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypropazine, methylephedrine, methylegonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, pimindine, pimozone, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocinide, tolpropamine, tolycaine, tranlylcypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleminamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane, I.; McKinnon, A.; Flanagan, R. J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, *323*, 191-225.

Perindopril

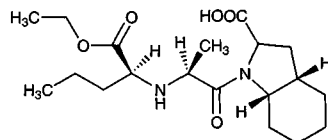
Molecular formula: C₁₉H₃₂N₂O₅

Molecular weight: 368.47

CAS Registry No.: 82834-16-0, 107133-36-8 (erbumine salt)

Merck Index: 7311

Lednicer No.: 5 111



SAMPLE

Matrix: blood, urine

Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 µL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) µL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

HPLC VARIABLES

Guard column: 20 mm long Symmetry C18

Column: 250 × 4.6 5 µm Symmetry C8 (Waters)

Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.

Column temperature: 30

Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)

Injection volume: 10-30

Detector: UV 206.4

CHROMATOGRAM

Retention time: 13.698

KEY WORDS

whole blood

REFERENCE

Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149-163.

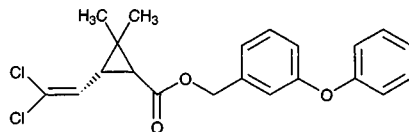
Permethrin

Molecular formula: C₂₁H₂₀Cl₂O₃

Molecular weight: 391.29

CAS Registry No.: 52645-53-1

Merck Index: 7321



SAMPLE

Matrix: blood, tissue

Sample preparation: Homogenize (Labsonic 2000 U sonicator with titanium probe) sciatic nerve or brain with 2 mL water. Homogenize (Stomacher Lab Blender) liver samples. Shake plasma or tissue homogenate with 8 mL n-pentane, centrifuge, repeat twice more. Combine the organic layers and evaporate them to dryness under a stream of nitrogen at 30°, reconstitute the residue in 500 µL MeOH, inject an aliquot.

HPLC VARIABLES

Column: 120 × 4 5 µm Nucleosil 120 C18

Mobile phase: MeOH:water:chloroform:acetic acid 54.5:39.5:5:1

Flow rate: 1.1

Detector: UV 254

CHROMATOGRAM

Limit of quantitation: 200 ng/mL (tissue), 100 ng/mL (plasma)

OTHER SUBSTANCES

Extracted: metabolites

KEY WORDS

rat; plasma; brain; sciatic nerve; pharmacokinetics

REFERENCE

Anadón,A.; Martínez-Larrañaga,M.R.; Díaz,M.J.; Bringas,P. Toxicokinetics of permethrin in the rat, *Toxicol.Appl.Pharmacol.*, **1991**, 110, 1–8.

SAMPLE

Matrix: bulk

Sample preparation: Dissolve the compound in benzene. Inject an aliquot. (Protect the sample from light! Caution! Benzene is a carcinogen!)

HPLC VARIABLES

Column: 250 × 4 10 µm Lichrospher Si60

Mobile phase: Hexane:benzene 50:50

Flow rate: 1

Detector: UV 280; Polarimeter, diode-laser polarimetric Chiral Monitor 2000 (Applied Chromatography System, Great Britain) collimated laser diode 30 mW at 830 nm, flow cell 4.8 cm, volume 73 µL

CHROMATOGRAM

Retention time: 3.63 (cis 1RS, 3SR), 4.39 (trans 1RS, 3RS) (UV detection); 3.76 (cis 1R, 3S [+]), 4.03 (cis 1S, 3R [-]), 4.52 (trans 1R, 3R [+]), 4.85 (trans 1S, 3S [-]) (polarimetric detection)

Limit of detection: 91 µg

OTHER SUBSTANCES

Simultaneous: cypermethrin, deltamethrin

KEY WORDS

chiral; normal phase

REFERENCE

Díaz,A.N.; Sánchez,F.G.; Pareja,A.G. Resolution of deltamethrin, permethrin, and cypermethrin enantiomers by high-performance liquid chromatography with diode-laser polarimetric detection, *J.Chromatogr.Sci.*, **1998**, 36, 210–216.

SAMPLE

Matrix: formulations

Sample preparation: Dilute formulation 100-fold with MeOH, centrifuge at 1250 g for 10 min, inject a 10 µL aliquot of the supernatant.

HPLC VARIABLES

Column: 30 × 4.6 3 µm P-E 3 × 3 C18 (Perkin-Elmer)

Mobile phase: MeCN:water 85:15

Flow rate: 2

Injection volume: 10

Detector: UV 229

CHROMATOGRAM

Limit of detection: 400 pg

OTHER SUBSTANCES

Also analyzed: amitraz (UV 313), chlorpyrifos (UV 313), coumaphos (UV 313), crotoxyphos (UV 229), phosmet (UV 229)

REFERENCE

Rice,L.G. Rapid separation of pesticides by high-performance liquid chromatography with 3-µm columns, *J.Chromatogr.*, **1984**, 317, 523–526.

SAMPLE

Matrix: fruit, vegetables

Sample preparation: Prepare a cleanup column by placing 4 g Florisil, 1 g activated charcoal, and a 20 mm layer of anhydrous sodium sulfate in a 400 × 10 glass column, wash with 40 mL toluene, wash with 40 mL toluene:MeCN 99:1. Homogenize 25 g chopped fruit or vegetable with 70 mL MeOH at high speed for 3 min, filter, homogenize solid with 30 mL MeOH, filter. Combine the filtrates and add them to 60 mL toluene and 300 mL 10% NaCl in water, shake well for 3 min, let layers separate. Dry the organic layer by passing it through 20 g anhydrous sodium sulfate in a 20 mm diameter column, concentrate to about 5 mL under reduced pressure at 80°, add to the cleanup column, elute with 40 mL toluene:MeCN 99:1. Evaporate the eluate just to dryness under reduced pressure at 80°, reconstitute with 1 mL MeOH, inject an aliquot. (Reflux activated charcoal (20-40 mesh) with 1 M HCl for 4 h, wash with water until the washings are neutral, dry at 95-100° (J.Assoc.Off.Anal.Chem. 1983, 66, 1013). Heat 60-100 mesh Florisil at 200° for 24 h, cool, add 4% water, mix thoroughly, store in a sealed jar (J.Assoc.Off.Anal.Chem. 1983, 66, 1003).)

HPLC VARIABLES

Column: 300 × 3.9 10 µm µBondapak C18

Mobile phase: Gradient. MeCN:water from 62:38 to 78:22 over 32 min (Waters curve 6).

Column temperature: 50

Flow rate: 1.5

Detector: UV 206

CHROMATOGRAM

Retention time: 24.60, 27.01

Limit of detection: 50 ng/g

OTHER SUBSTANCES

Simultaneous: allethrin, biphenthrin, cypermethrin, fenpropathrin, fenvalerate, flucythrinate, methothrin, Py-115, tetramethrin

KEY WORDS

cucumber; tomato; cabbage; apple; pear; peach; SPE

REFERENCE

Pang,G.-F.; Chao,Y.-Z.; Liu,X.-S.; Fan,C.-L. Multiresidue liquid chromatographic method for simultaneous determination of pyrethroid insecticides in fruits and vegetables, *J.AOAC Int.*, **1995**, 78, 1474-1480.

SAMPLE

Matrix: rice

Sample preparation: 30 g Rice + 50 mL acetone, let stand with occasional shaking for 48 h, inject a 10 µL aliquot.

HPLC VARIABLES

Guard column: Guard Pak

Column: 150 × 3.9 Novapak C18

Mobile phase: MeCN:water 75:25

Flow rate: 1

Injection volume: 10

Detector: UV 225

CHROMATOGRAM

Retention time: 5.5

Limit of detection: 1 µg/g

OTHER SUBSTANCES

Extracted: bioremethrin, deltamethrin, fenvalerate, phenothrin, piperonyl butoxide

REFERENCE

Haddad,P.R.; Brayan,J.G.; Sharp,G.J.; Dilli,S.; Desmarchelier,J.M. Determination of pyrethroid residues on paddy rice by reversed-phase high-performance liquid chromatography, *J.Chromatogr.*, **1989**, 461, 337-346.

SAMPLE**Matrix:** solutions

HPLC VARIABLES**Guard column:** 50 × 4 40 µm pellicular material**Column:** 250 × 4.6 5 µm silica (IBM)**Mobile phase:** Hexane:dichloromethane:isopropanol 99:1:0.07**Flow rate:** 1**Injection volume:** 10**Detector:** UV 254

CHROMATOGRAM**Retention time:** k' 4.86 (cis), k' 6.68 (trans)

OTHER SUBSTANCES**Also analyzed:** allethrin, chrysanthemol, dimethrin, ethyl chrysanthemate, cyfluthrin (bay-throid), phenothrin, resmethrin, RU-11679, tetramethrin

KEY WORDSnormal phase

REFERENCEAbidi, S.L. Column selectivity in high-performance liquid chromatography of substituted *gem*-dimethylcyclopropanes, *J.Chromatogr.*, **1986**, 368, 59–76.

SAMPLE**Matrix:** solutions

HPLC VARIABLES**Column:** 125 × 4.6 PartiSphere Silica (Whatman)**Mobile phase:** Hexane:dichloromethane 99:1**Flow rate:** 1**Detector:** UV 270

CHROMATOGRAM**Retention time:** 1.57 (cis), 2.17 (trans)

KEY WORDSnormal phase

REFERENCE*Baxter Scientific Products Catalog*, 1990-1, p. 173.

SAMPLE**Matrix:** solutions**Sample preparation:** Inject an aliquot of a 0.1-1 mg/mL solution in hexane.

HPLC VARIABLES**Guard column:** 5 µm Spherisorb NH₂**Column:** 250 × 4.6 Pirkle ionic type 1-A column (Technicol)**Mobile phase:** Hexane:isopropanol 99.95:0.05**Flow rate:** 0.8**Detector:** UV 230

CHROMATOGRAM**Retention time:** 18.2, 18.8, 26.5, 27.1 (enantiomers)

OTHER SUBSTANCES**Also analyzed:** phenothrin, resmethrin

KEY WORDS

chiral

REFERENCE

Lisseter, S.G.; Hambling, S.G. Chiral high-performance liquid chromatography of synthetic pyrethroid insecticides, *J.Chromatogr.*, **1991**, 539, 207–210.

SAMPLE**Matrix:** solutions

Sample preparation: Inject 100 mL river water on to column A at 5 mL/min and let the effluent flow to waste, backflush the contents of column A on to column B and start the gradient, monitor the effluent from column B. At the end of each run backflush column A with 5 mL water, with 30 mL 100 mM pH 2 sodium citrate buffer, with 10 mL water, with 5 mL MeCN, and with 10 mL hexane:dichloromethane 50:50. Wash new column A with 10 mL water.

HPLC VARIABLES

Column: A 20 × 3 10 µm PRP-1 (Hamilton); B 30 × 4.6 10 µm RP-18 (Brownlee) + 250 × 4.6 5 µm LiChrospher C18

Mobile phase: Gradient. MeCN:water from 5:95 to 90:10 over 1 h, maintain at 90:10 for 4 min, return to initial conditions (?) over 1 min, re-equilibrate for 10 min.

Flow rate: 1 for 64 min, to 1.5 over 1 min, maintain at 1.5 for 5 min, to 1 over 5 min

Injection volume: 100000

Detector: UV 210

CHROMATOGRAM

Retention time: 62.2 (trans), 63.6 (cis)

Limit of detection: 100 ng/L

OTHER SUBSTANCES

Simultaneous: alachlor, aldicarb, aldicarb oxime, atrazine, carbofuran, chlorobenzilate, chlorothalonil, chlorpyrifos methyl, chlortoluron, cypermethrin, DDT, deltamethrin, diazinon, diclofop methyl, dimethoate, diuron, ethofumesate, fenitrothion, fenvalerate, fluzifop butyl, fluometuron, linuron, metalaxyl, metamitron, methomyl, metobromuron, metolachlor, molinate, oxamyl, paraoxon, paraoxon methyl, parathion, parathion methyl, pendimethalin, phenmediphan, pirimphos, pirimphos methyl, prometryne, propanil, propiconazole, simazine, terbuthylazine, trifluraline

Interfering: p,p'-DDE

KEY WORDS

river water; column-switching

REFERENCE

Papadopoulos-Mourkidou, E.; Patsias, J. Development of a semi-automated high-performance liquid chromatographic-diode array detection system for screening pesticides at trace levels in aquatic systems of the Axios River basin, *J.Chromatogr.A*, **1996**, 726, 99–113.

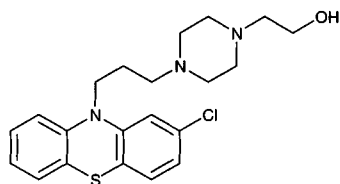
Perphenazine

Molecular formula: C₂₁H₂₆ClN₃OS

Molecular weight: 403.98

CAS Registry No.: 58-39-9

Merck Index: 7323

**SAMPLE****Matrix:** blood

Sample preparation: 3 mL Serum + 50 ng thiethylperazine maleate + 3 mL diluted Titrisol (pH 10 borate buffer, Merck) + 4 mL heptane:isoamyl alcohol 97:3, shake thoroughly for 15 s,

centrifuge at 2500 g for 5 min. Remove the organic phase and add it to 1.5 mL 50 mM sulfuric acid containing 0.1% Na₂S₂O₃, mix for 15 s, centrifuge at 2500 g for 10 min. Remove the aqueous phase. Repeat the extraction and back extraction. Combine the aqueous phases and add them to 1.5 mL 2 M pH 9.1 glycine buffer, add 200 µL n-hexane:isoamyl alcohol 97:3, vortex for 25 s. Remove the organic phase and evaporate it to dryness in a desiccator, reconstitute in mobile phase, inject an aliquot.

HPLC VARIABLES

Column: 300 × 3.9 10 µm µBondapak C18

Mobile phase: MeCN:water:acetic acid 65:35:3 containing 10 mM dodecyl hydrogen sulfate

Flow rate: 2

Detector: UV 257

CHROMATOGRAM

Retention time: 8.5

Internal standard: thiethylperazine (13)

Limit of detection: 0.3 ng/mL

OTHER SUBSTANCES

Extracted: metabolites

Noninterfering: alimemazine, biperidine, carbamazepine, chlorpromazine, clomipramine, diazepam, dihydroergotamine, disulfiram, dixyrazine, haloperidol, levomepromazine, nitrazepam, orphenadrine, promethazine, propiomazine, thioridazine, trimipramine, vitamins

KEY WORDS

serum

REFERENCE

Larsson,M.; Forsman,A. A high-performance liquid chromatographic method for the assay of perphenazine and its dealkylated metabolite in serum after therapeutic doses, *Ther.Drug Monit.*, **1983**, 5, 225–228.

SAMPLE

Matrix: blood

Sample preparation: 2.5 mL Plasma + 7.5 ng IS + 100 µL 1 M NaOH + 6 mL ethyl acetate: hexane 2:1, shake vigorously for 30 s, centrifuge for 3 min. Remove the organic phase and add it to 2 mL 100 mM HCl, shake vigorously for 30 s, centrifuge for 3 min. Remove the aqueous phase and add it to 100 µL 6 M NaOH, add 3 mL hexane, shake vigorously for 30 s. Remove the organic layer and evaporate it to dryness under a stream of nitrogen, reconstitute the residue in 75 µL mobile phase, inject a 20 µL aliquot.

HPLC VARIABLES

Column: 20 × 4.6 3 µm C18 (Perkin-Elmer)

Mobile phase: MeOH:water:dichloromethane:ammonia 200:40:10:3

Flow rate: 1

Injection volume: 20

Detector: UV 257

CHROMATOGRAM

Retention time: 2.1

Internal standard: 4-[3-(2,8-dichlorophenothiazin-10-yl)propyl]-1-piperazinethanol(2.5)

Limit of quantitation: 0.5 nM

OTHER SUBSTANCES

Extracted: metabolites

Noninterfering: amitriptyline, biperidine, clomipramine, disulfiram, imipramine, levomepromazine, nitrazepam, nortriptyline, orphenadrine

Interfering: diazepam

KEY WORDS

plasma

REFERENCE

Larsen,N.-E.; Hansen,L.B.; Knudsen,P. Quantitative determination of perphenazine and its dealkylated metabolite using high-performance liquid chromatography, *J.Chromatogr.*, **1985**, *341*, 244-250.

SAMPLE

Matrix: blood

Sample preparation: 1 mL Plasma + 10 μ L 1 mg/mL prochlorperazine in 25 mM pH 2.4 KH_2PO_4 , adjust pH to 9.0 with 1 M NaOH, add 6 mL n-hexane:ethyl acetate 1:2, shake for 30 s, centrifuge at 3000 g for 3 min. Remove the organic layer and add it to 100 μ L pH 2.4 KH_2PO_4 , shake for 30 s, centrifuge at 3000 g for 3 min, inject a 50 μ L aliquot of the organic layer.

HPLC VARIABLES

Column: 120 \times 4.6 5 μ m Nucleosil C18

Mobile phase: MeCN:MeOH:buffer 26:4:70 (Buffer was 10 mM KH_2PO_4 containing 5 mM tetramethylammonium chloride adjusted to pH 2.4 with 85% phosphoric acid.)

Flow rate: 1

Injection volume: 50

Detector: E, ESA Coulochem Model 5100A, detector 1 +0.20 V, detector 2 +0.73 V, guard cell 0.75 V

CHROMATOGRAM

Retention time: 15.01

Internal standard: prochlorperazine dimaleate (17.80)

Limit of quantitation: 0.5 ng/mL

OTHER SUBSTANCES

Extracted: metabolites

Simultaneous: alprazolam, chlorpheniramine, diltiazem, lorazepam, mesoridazine, nifedipine, ranitidine

Noninterfering: doxepin, metoprolol, nordoxepin, nortriptyline, propranolol, theophylline, tri-fluoperazine, trihexyphenidyl, verapamil

KEY WORDS

plasma; protect from light

REFERENCE

Foglia,J.P.; Sorisio,D.; Kirshner,M.A.; Mulsant,B.H.; Perel,J.M. Quantitative determination of perphenazine and its metabolites in plasma by high-performance liquid chromatography and coulometric detection, *J.Chromatogr.B*, **1995**, *668*, 291-297.

SAMPLE

Matrix: blood, urine

Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 μ L MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) μ L aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

HPLC VARIABLES

Guard column: 20 mm long Symmetry C18

Column: 250 \times 4.6 5 μ m Symmetry C8 (Waters)

Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.

Column temperature: 30

Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)

Injection volume: 10-30

Detector: UV 255.8

CHROMATOGRAM**Retention time:** 15.96

KEY WORDSwhole blood

REFERENCE

Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, 763, 149–163.

SAMPLE**Matrix:** formulations

Sample preparation: Add 5 mL water to powdered tablets containing 4 mg perphenazine, heat on a water-bath for 3 min. Cool, add 50 mL water, shake for 15 min and make up to 100 mL with MeOH. Filter, remove a 5 mL aliquot, add IS to a concentration of 3 µg/mL, make up to 50 mL with mobile phase, inject an aliquot.

HPLC VARIABLES**Column:** 150 × 3.9 Novapak-phenyl-4**Mobile phase:** MeOH:15 mM pH 6.5 sodium acetate buffer 81:19**Flow rate:** 1.0**Injection volume:** 10**Detector:** UV 254

CHROMATOGRAM**Retention time:** 4.4**Internal standard:** pindolol (2.5)**Limit of detection:** 100 pg

OTHER SUBSTANCES**Simultaneous:** degradation products

KEY WORDStablets

REFERENCE

Al-Obaid,A.M.; Hagga,M.E.M.; El-Khawad,I.E.; El-Mahi,O.H.M. Simultaneous quantitation of some phenothiazine drug substances and their monosulphoxide degrades by high performance liquid chromatography (HPLC), *J.Liq.Chromatogr.Rel.Technol.*, **1996**, 19, 1369–1389.

SAMPLE**Matrix:** formulations

Sample preparation: Syrup, injections. Measure amount of syrup equivalent to about 5 mg perphenazine, add 5 mL 0.5 mg/mL trifluoperazine hydrochloride in MeOH, make up to 50 mL with MeOH, mix, inject a 10 µL aliquot. Tablets. Finely powder tablets, weigh out amount equivalent to 10 mg perphenazine, add 20 mL 1% HCl, shake for 20 min, centrifuge. Remove a 10 mL aliquot and add it to 5 mL 0.5 mg/mL trifluoperazine hydrochloride in MeOH, make up to 50 mL with MeOH, mix, inject a 10 µL aliquot.

HPLC VARIABLES**Column:** 150 × 4.6 5 µm Zorbax CN**Mobile phase:** MeCN:MeOH:25 mM pH 4.5 acetate buffer 40:30:30**Flow rate:** 2.5**Injection volume:** 10**Detector:** UV 229

CHROMATOGRAM**Retention time:** 4.6**Internal standard:** trifluoperazine (6)

OTHER SUBSTANCES

Simultaneous: perphenazine sulfoxide, degradation products

KEY WORDS

tablets; syrup; injections

REFERENCE

Beaulieu, N.; Lovering, E. G. Liquid chromatographic method for perphenazine and its sulfoxide in pharmaceutical dosage forms for determination of stability, *J. Assoc. Off. Anal. Chem.*, **1986**, 69, 167-169.

SAMPLE

Matrix: solutions

Sample preparation: Prepare a 10 µg/mL solution in MeOH, inject a 20 µL aliquot.

HPLC VARIABLES

Column: 125 × 4.9 Spherisorb S5W silica

Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

Flow rate: 2

Injection volume: 20

Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM

Retention time: 2.6

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzotamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazepine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipranone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserine, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamlamine, meclophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypropazine, methylephedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, naltrexone, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscipine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, phenadoxone, phenampromide, phenazocine, phenbutazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, pimindine, pimizide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, propidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocanide, tolpropamine, tolycaine, tranlycypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleminamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane,I.; McKinnon,A.; Flanagan,R.J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J.Chromatogr.*, **1985**, 323, 191–225.

SAMPLE

Matrix: solutions

Sample preparation: Dissolve in MeOH:water 1:1 at a concentration of 50 µg/mL, inject a 10 µL aliquot.

HPLC VARIABLES

Column: 300 × 3.9 10 µm µBondapak C18

Mobile phase: MeOH:acetic acid:triethylamine:water 70:1.5:0.5:28

Flow rate: 1.5

Injection volume: 10

Detector: UV

CHROMATOGRAM

Retention time: k' 1.59

REFERENCE

Roos,R.W.; Lau-Cam,C.A. General reversed-phase high-performance liquid chromatographic method for the separation of drugs using triethylamine as a competing base, *J.Chromatogr.*, **1986**, 370, 403–418.

SAMPLE

Matrix: solutions

Sample preparation: Prepare a 1 mg/mL solution in MeOH, inject a 5 µL aliquot.

HPLC VARIABLES

Column: 250 × 4.6 5 µm Lichrosphere cyanopropyl

Mobile phase: Carbon dioxide:MeOH:isopropylamine 90:10:0.05

Column temperature: 50

Flow rate: 3

Injection volume: 5

Detector: UV 220

CHROMATOGRAM

Retention time: 3.75

OTHER SUBSTANCES

Simultaneous: benactyzine, buclizine, hydroxyzine, thioridazine, amitriptyline, desipramine, imipramine, nortriptyline, protriptyline

KEY WORDS

SFC; pressure 200 bar

REFERENCE

Berger,T.A.; Wilson,W.H. Separation of drugs by packed column supercritical fluid chromatography. 2. Anti-depressants, *J.Pharm.Sci.*, **1994**, 83, 287–290.

SAMPLE

Matrix: solutions

Sample preparation: Prepare a 1 mg/mL solution in MeOH, inject a 5 µL aliquot.

HPLC VARIABLES

Column: 250 × 4.6 5 µm Lichrosphere cyanopropyl

Mobile phase: Carbon dioxide:MeOH:isopropylamine 94:6:0.03

Column temperature: 50

Flow rate: 3

Injection volume: 5

Detector: UV 254

CHROMATOGRAM

Retention time: 6.0

OTHER SUBSTANCES

Simultaneous: triflupromazine, carphenazine, methotrimeprazine, promazine, chlorprothixene, deserpidine, thiothixene, reserpine

Also analyzed: acetophenazine, ethopropazine, promethazine, propiomazine

KEY WORDS

SFC; pressure 200 bar

REFERENCE

Berger, T.A.; Wilson, W.H. Separation of drugs by packed column supercritical fluid chromatography. 1. Pheno-thiazine antipsychotics, *J.Pharm.Sci.*, **1994**, 83, 281–286.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 5 μ m Diol (Keystone)

Mobile phase: Carbon dioxide:MeOH containing 1 mM t-butylammonium hydroxide

Flow rate: 0.5 (carbon dioxide), 0.1 (MeOH/t-butylammonium hydroxide)

Detector: UV 254

CHROMATOGRAM

Retention time: 6

OTHER SUBSTANCES

Simultaneous: impurities

KEY WORDS

outlet pressure 3500 psi; SFC; back-pressure regulator heated to 60°

REFERENCE

Ashraf-Khorassani, M.; Levy, J.M. Addition of modifier in supercritical fluid chromatography using a microbore reciprocating pump, *Chromatographia*, **1995**, 40, 78–84.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 \times 4.6 5 μ m Supelcosil LC-DP (A) or 250 \times 4 5 μ m LiChrospher 100 RP-8 (B)

Mobile phase: MeCN:0.025% phosphoric acid:buffer 25:10:5 (A) or 60:25:15 (B) (Buffer was 9 mL concentrated phosphoric acid and 10 mL triethylamine in 900 mL water, adjust pH to 3.4 with dilute phosphoric acid, make up to 1 L.)

Flow rate: 0.6

Injection volume: 25

Detector: UV 229

CHROMATOGRAM

Retention time: 13.16 (A), 6.33 (B)

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetaminophen, acetazolamide, acetophenazine, albuterol, alprazolam, amitriptyline, amobarbital, amoxapine, antipyrine, atenolol, atropine, azatadine, baclofen, benzocaine, bromocriptine, brompheniramine, brotizolam, bupivacaine, buspirone, butabarbital, butalbital, caffeine, carbamazepine, cetirizine, chlorcyclizine, chlordi-azepoxide, chlormezanone, chloroquine, chlorpheniramine, chlorpromazine, chlorpropamide,

chlorprothixene, chlorthalidone, chlorzoxazone, cimetidine, cisapride, clomipramine, clonazepam, clonidine, clozapine, cocaine, codeine, colchicine, cyclizine, cyclobenzaprine, dantrolene, desipramine, diazepam, diclofenac, diflunisal, diltiazem, diphenhydramine, diphenidol, diphenoxylate, dipyridamole, disopyramide, dobutamine, doxapram, doxepin, droperidol, encainide, ethidium bromide, ethopropazine, fenoprofen, fentanyl, flavoxate, fluoxetine, fluphenazine, flurazepam, flurbiprofen, fluvoxamine, furosemide, glutethimide, glyburide, guaifenesin, haloperidol, homatropine, hydralazine, hydrochlorothiazide, hydrocodone, hydromorphone, hydroxychloroquine, hydroxyzine, ibuprofen, imipramine, indomethacin, ketoconazole, ketoprofen, ketorolac, labetalol, levorphanol, lidocaine, loratadine, lorazepam, lovastatin, loxapine, mazinol, mefenamic acid, meperidine, mephenytoin, mepivacaine, mesoridazine, metaproterenol, metformin, methadone, methdilazine, methocarbamol, methotrexate, methotrimetaprazine, methoxamine, methylodopa, methylphenidate, metoclopramide, metolazone, metoprolol, metronidazole, midazolam, moclobemide, morphine, nadolol, nalbuphine, naloxone, naphazoline, naproxen, nifedipine, nizatidine, norepinephrine, nortriptyline, oxazepam, oxycodone, oxymetazoline, paroxetine, pemoline, pentazocine, pentobarbital, pentoxifylline, pheniramine, phenobarbital, phenol, phenolphthalein, phentolamine, phenylbutazone, phenyltoloxamine, phenytoin, pimozide, pindolol, piroxicam, pramoxine, prazepam, prazosin, probenecid, procainamide, procaine, prochlorperazine, procyclidine, promazine, promethazine, propafenone, propantheline, propiomazine, propofol, propranolol, protriptyline, quazepam, quinidine, quinine, racemethorphan, ranitidine, remoxipride, risperidone, salicylic acid, scopolamine, secobarbital, sertraline, sotalol, spironolactone, sulfinpyrazone, sulindac, temazepam, terbutaline, terfenadine, tetracaine, theophylline, thiethylperazine, thiopental, thioridazine, thiothixene, timolol, tocainide, tolbutamide, tolmetin, trazodone, triamterene, triazolam, trifluoperazine, triflupromazine, trimeprazine, trimethoprim, trimipramine, verapamil, warfarin, xylometazoline, yohimbine, zopiclone

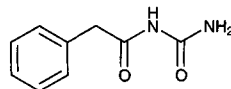
KEY WORDS

details of plasma extraction

REFERENCE

Koves, E.M. Use of high-performance liquid chromatography-diode array detection in forensic toxicology, *J.Chromatogr.A*, **1995**, 692, 103-119.

Phenacemide



Molecular formula: $C_9H_{10}N_2O_2$

Molecular weight: 178.19

CAS Registry No.: 63-98-9

Merck Index: 7343

Lednicer No.: 1 95

SAMPLE

Matrix: blood

Sample preparation: Prepare an SPE cartridge by plugging the end of a 1 mL disposable pipette tip with glass wool and adding about 100 mg Chromosorb P/NAW. Add 50 μ L plasma then 50 μ L 10 μ g/mL tolylphenobarbital in 200 mM HCl to the SPE cartridge, let stand for 2 min, elute with 1 mL chloroform:isopropanol 6:1. Evaporate the eluate to dryness under a stream of nitrogen at 30°, reconstitute the residue in 100 μ L mobile phase, inject a 15 μ L aliquot.

HPLC VARIABLES

Column: 150 \times 4.6 5 μ m Supelcosil-LC-8

Mobile phase: MeCN:water 20:80

Flow rate: 3.3

Injection volume: 15

Detector: UV 208

CHROMATOGRAM

Retention time: 2.36

Internal standard: tolylphenobarbital (7.57)

Limit of detection: 50-100 ng/mL

OTHER SUBSTANCES

Extracted: theophylline, caffeine, barbital, ethosuximide, primidone, carbamazepinediol, nirvanol, phenobarbital, chloramphenicol, butabarbital, carbamazepine epoxide, mephénytoin, pentobarbital, amobarbital, carbamazepine, glutethimide, phénytoin, secobarbital, methaqualone

Noninterfering: acetaminophen, amikacin, amitriptyline, clonazepam, cyclosporine, desipramine, diazepam, digoxin, disopyramide, gentamicin, imipramine, lidocaine, methotrexate, N-acetylprocainamide, netilmicin, nortriptyline, procainamide, quinidine, salicylic acid, sulfamethoxazole, tobramycin, trimethoprim, valproic acid, p-hydroxyphenobarbital, vancomycin

Interfering: methyprylon

KEY WORDS

plasma; SPE

REFERENCE

Svinarov, D.A.; Dotchev, D.C. Simultaneous liquid-chromatographic determination of some bronchodilators, anticonvulsants, chloramphenicol, and hypnotic agents, with Chromosorb P columns used for sample preparation, *Clin.Chem.*, **1989**, *35*, 1615-1618.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 100 × 4.6 3 µm 208HS3410 (Vydac)

Mobile phase: Gradient. MeCN:water from 15:85 to 60:40 over 10 min.

Flow rate: 1.5

Detector: UV 210 (?)

CHROMATOGRAM

Retention time: 2.8

OTHER SUBSTANCES

Simultaneous: barbital, carbamazepine, diazepam, ethotoin, mephénytoin, methsuximide, phenobarbital, phensuximide

REFERENCE

Vydac HPLC Catalog, 1994-5, **1994**, p. 26.

Phenacetin

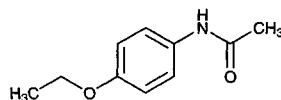
Molecular formula: C₁₀H₁₃NO₂

Molecular weight: 179.22

CAS Registry No.: 62-44-2

Merck Index: 7344

Lednicer No.: 1 111



SAMPLE

Matrix: solution

HPLC VARIABLES

Guard column: 20 × 2 pellicular C18 (Alltech)

Column: 300 × 3.9 10 µm µBondapak C18

Mobile phase: MeCN:THF:100 mM ammonium acetate 22.5:5.5:72

Flow rate: 1.0

Injection volume: 50

Detector: UV 283

CHROMATOGRAM

Retention time: 10.9

OTHER SUBSTANCES

Simultaneous: chlorzoxazone

REFERENCE

Frye,R.F.; Stiff,D.D. Determination of chlorzoxazone and 6-hydroxychlorzoxazone in human plasma and urine by high-performance liquid chromatography, *J.Chromatogr.B*, **1996**, 686, 291–296.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 300 × 3.9 µBondapak C18

Mobile phase: MeCN:MeOH:10 mM phosphate buffer 22.5:37.5:40

Flow rate: 0.8

Detector: UV 220

CHROMATOGRAM

Retention time: 5.4

OTHER SUBSTANCES

Simultaneous: 4-hydroxy-midazolam, α-hydroxy-midazolam, midazolam

REFERENCE

von Moltke,L.L.; Greenblatt,D.J.; Schmider,J.; Duan,S.X.; Wright,C.E.; Harmatz,J.S.; Shader,R.I. Midazolam hydroxylation by human liver microsomes in vitro: Inhibition by fluoxetine, norfluoxetine, and by azole antifungal agents, *J.Clin.Pharmacol.*, **1996**, 36, 783–791.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 × 4.6 Zorbax RX

Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.

Column temperature: 30

Flow rate: 2

Detector: UV 210

OTHER SUBSTANCES

Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitriptyline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepam, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlor-diazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisolone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fen-

camfamine, fenpropofen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiaicol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, iminostilbene, imipramine, indomethacin, isocarboxtyril, isocarboxazid, isoniazid, isoproterenol, isoxsuprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephenytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, methapyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyl dopa, methyl dopamine, methylphenidate, methylprednisolone, methyltestosterone, methylprylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, oxymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, phenazocine, phenazopyridine, phenacyclidine, phendimetrazine, phenelzine, pheniramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrilamine, pyrrithyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sulfadiazine, sulfadimethoxine, sulfaethidole, sulfamerazine, sulfamethazine, sulfamethoxizole, sulfanilamide, sulfapyridine, sulfasoxizole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranlycypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripelennamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE

Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233–242.

Phenadoxone

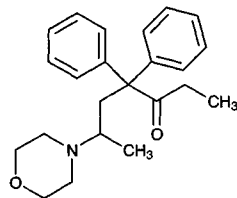
Molecular formula: $C_{23}H_{29}NO_2$

Molecular weight: 351.49

CAS Registry No.: 467-84-5, 545-91-5 (HCl)

Merck Index: 7348

Lednicer No.: 1 80



SAMPLE

Matrix: solutions

Sample preparation: Prepare a 10 µg/mL solution in MeOH, inject a 20 µL aliquot.

HPLC VARIABLES

Column: 125 × 4.9 Spherisorb S5W silica

Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

Flow rate: 2

Injection volume: 20

Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM

Retention time: 1.35

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazepine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipiprone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscyne, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserine, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamylamine, meclorphenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylegonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazoline, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, pimindine, pimizide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranlycypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleminamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane, I.; McKinnon, A.; Flanagan, R. J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, 323, 191–225.

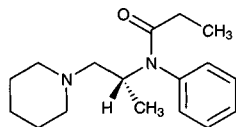
Phenampromide

Molecular formula: C₁₇H₂₆N₂O

Molecular weight: 274.41

CAS Registry No.: 129-83-9

Merck Index: 7353



SAMPLE

Matrix: solutions

Sample preparation: Prepare a 10 µg/mL solution in MeOH, inject a 20 µL aliquot.

HPLC VARIABLES

Column: 125 × 4.9 Spherisorb S5W silica

Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

Flow rate: 2

Injection volume: 20

Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM

Retention time: 2.2

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipiprone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserine, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamlamine, meclophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylphenedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenylglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, pimindine, pimizide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazacine, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocanide, tolpropamine, tolycaine, tranlycypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleennamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane, I.; McKinnon, A.; Flanagan, R.J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, *323*, 191–225.

Phenazocine

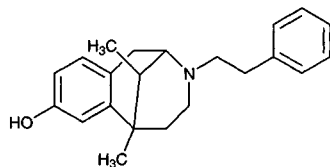
Molecular formula: C₂₂H₂₇N

Molecular weight: 321.46

CAS Registry No.: 127-35-5, 1239-04-9 (±-hydrobromide)

Merck Index: 7360

Lednicer No.: 1 298



SAMPLE

Matrix: solutions

Sample preparation: Prepare a 10 µg/mL solution in MeOH, inject a 20 µL aliquot.

HPLC VARIABLES

Column: 125 × 4.9 Spherisorb S5W silica

Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

Flow rate: 2

Injection volume: 20

Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM

Retention time: 2.1

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzocetamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazepine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipanone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupentixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserin, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamlamine, meclorphenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylegonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, pimindine, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocanide, tolpropamine, tolycaine, tranlylcypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripeleminamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane, I.; McKinnon, A.; Flanagan, R. J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, *323*, 191–225.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 × 4.6 Zorbax RX

Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200

mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.

Column temperature: 30

Flow rate: 2

Detector: UV 210

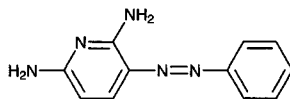
OTHER SUBSTANCES

Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitrityline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepam, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlor-diazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fen-camfamine, fenopropfen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiaicol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, imino-stilbene, imipramine, indomethacin, isocarboxystyryl, isocarboxazid, isoniazid, isoproterenol, isox-suprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephenytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, meth-apyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyl dopa, methyl dopamine, methylphenidate, methylprednisolone, methyl-testosterone, methyprylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, ox-ymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenazopyridine, phencyclidine, phendimetrazine, phenelzine, phen-iramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenyl-ephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyri-lamine, pyrithyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfa-merazine, sulfamethazine, sulfamethoxazole, sulfanilamide, sulfapyridine, sulfasoxazole, sulin-dac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, the-obromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranlycy-promine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripeleonnamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE

Hill, D.W.; Kind, A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J. Anal. Toxicol.*, **1994**, *18*, 233–242.

Phenazopyridine



Molecular formula: $C_{11}H_{11}N_5$

Molecular weight: 213.24

CAS Registry No.: 94-78-0, 136-40-3 (HCl)

Merck Index: 7361

Lednicer No.: 1 255

SAMPLE

Matrix: formulations

Sample preparation: Dissolve a capsule in 1 L 100 mM HCl, inject a 20 μ L aliquot.

HPLC VARIABLES

Column: 250 \times 4.6 7 μ m Zorbax TMS

Mobile phase: MeCN:isopropanol:acetic acid:water 30:30:1:39

Flow rate: 1.8

Injection volume: 20

Detector: UV 405

CHROMATOGRAM

Retention time: 5.25

OTHER SUBSTANCES

Noninterfering: sulfamethizole, tetracycline

KEY WORDS

capsules

REFERENCE

Du Preez, J.L.; Botha, S.A.; Lötter, A.P. High-performance liquid chromatographic determination of phenazopyridine hydrochloride, tetracycline hydrochloride and sulphamethizole in combination, *J. Chromatogr.*, **1985**, 333, 249–252.

SAMPLE

Matrix: formulations

Sample preparation: Powder tablets, add 40 mg trimethoprim, dissolve in 70 mL MeOH, filter (paper), wash filter with MeOH, make up filtrate to 100 mL with MeOH. Dilute a 1 mL aliquot to 10 mL, inject a 10 μ L aliquot.

HPLC VARIABLES

Column: 250 \times 4 10 μ m Nucleosil C18

Mobile phase: MeOH:MeCN:water:triethylamine 20:20:60:0.15, pH adjusted to 3.0 with phosphoric acid

Flow rate: 1.5

Injection volume: 10

Detector: UV 240

CHROMATOGRAM

Retention time: 7

Internal standard: trimethoprim (3)

Limit of quantitation: 10 μ g/mL

OTHER SUBSTANCES

Simultaneous: nalidixic acid

KEY WORDS

tablets

REFERENCE

Sane,R.T.; Ghadge,J.K.; Jani,A.B.; Vaidya,A.J.; Kotwal,S.S. Simultaneous high-performance liquid chromatographic determination of haloperidol with propantheline bromide, nalidixic acid with phenazopyridine hydrochloride, and dipyrindamole with aspirin in combined dosage (forms), *Indian Drugs*, **1992**, 29, 240-244.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 × 4.6 Zorbax RX

Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.

Column temperature: 30

Flow rate: 2

Detector: UV 210

OTHER SUBSTANCES

Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitriptyline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepam, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlor-diazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fencamfamine, fenpropofen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiaicol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, iminostilbene, imipramine, indomethacin, isocarboxtyril, isocarboxazid, isoniazid, isoproterenol, isoxsuprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephenytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, methapyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyl dopa, methyl dopamine, methylphenidate, methylprednisolone, methyltestosterone, methypylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscipine, nylidrin, oxazepam, oxycodone, oxymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phencyclidine, phendimetrazine, phenelzine, pheniramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrrolamine, pyrilthyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sulfadiazine, sulfadimethoxine, sulfaethidole, sulfamerazine, sulfamethazine, sulfamethoxazole, sulfanilamide, sulfapyridine, sulfasoxazole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranlycypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripeleennamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE

Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233–242.

SAMPLE

Matrix: urine

Sample preparation: 500 μ L Filtered (Millex-GS 0.22 μ m) urine + 500 μ L 200 mM pH 5 sodium acetate buffer + 3.75 μ L Glusulase + 12.5 μ g 2,6-diamino-3-(2-chlorophenylazo)pyridine + 20 μ g 3-acetaminophenol, heat at 37° for 16 h, freeze dry, reconstitute in 100 μ L water, add 900 μ L MeOH, sonicate, centrifuge. Remove the supernatant and evaporate it to dryness under a stream of nitrogen at 45°, reconstitute the residue in 500 μ L MeOH:MeCN:10 mM pH 6.5 ammonium formate buffer 37.5:37.5:25, sonicate, filter (Xydex LID/X ORG.2ns, 0.2 μ m PTFE membrane), inject a 20 μ L aliquot of the filtrate.

HPLC VARIABLES

Column: 250 \times 4.6 Supelcosil LC18 DB

Mobile phase: Gradient. A was MeCN:MeOH 50:50. B was 10 mM pH 6.5 ammonium formate buffer. A:B from 15:85 to 25:75 over 15 min, to 80:20 over 10 min.

Injection volume: 20

Detector: UV 430

CHROMATOGRAM

Retention time: 28.5

Internal standard: 2,6-diamino-3-(2-chlorophenylazo)pyridine (30.2), 3-acetaminophenol (10.4)

Limit of quantitation: 2 μ M

OTHER SUBSTANCES

Extracted: metabolites (UV 248 and UV 430)

KEY WORDS

rat; guinea pig; mouse; human

REFERENCE

Thomas,B.H.; Whitehouse,L.W.; Solomonraj,G.; Paul,C.J. Excretion of phenazopyridine and its metabolites in the urine of humans, rats, mice, and guinea pigs, *J.Pharm.Sci.*, **1990**, *79*, 321–325.

Phencyclidine

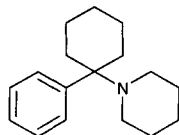
Molecular formula: C₁₇H₂₅N

Molecular weight: 243.39

CAS Registry No.: 77-10-1, 956-90-1 (HCl)

Merck Index: 7364

Lednicer No.: 1 56

**SAMPLE**

Matrix: blood

Sample preparation: 2 mL Whole blood or plasma + 2 mL buffer + 5 mL chloroform:isopropanol:n-heptane 60:14:26, shake gently horizontally for 10 min, centrifuge at 2800 g for 10 min. Remove the lower organic layer and evaporate it to dryness under vacuum at 45°, reconstitute the residue in 100 μ L mobile phase, centrifuge at 2800 g for 5 min, inject a 50 μ L aliquot of the supernatant. (Buffer was saturated ammonium chloride solution 25% diluted with water, adjusted to pH 9.5 with 25% ammonia solution.)

HPLC VARIABLES

Column: 300 \times 3.9 4 μ m NovaPack C18

Mobile phase: MeOH:THF:buffer 65:5:30 (Buffer was 0.68 g/L (10 mM (sic)) KH₂PO₄ adjusted to pH 2.6 with concentrated orthophosphoric acid.) (At the end of each session wash the column with water for 1 h and MeOH for 1 h, re-equilibrate for 30 min.)

Column temperature: 30

Flow rate: 0.8

Injection volume: 50

Detector: UV 262

CHROMATOGRAM

Retention time: 5.22

Limit of detection: <120 ng/mL

KEY WORDS

whole blood; plasma; interferences may occur—compounds (all of which are extracted) elute in this order: tenoxicam; iproniazid; methocarbamol; methotrexate; caffeine; nialamide; colchicine; cytarabine; benzoylecgonine; acetaminophen; diazoxide; dacarbazine; sulfinpyrazole; flumazenil; sulpride; morphine; atenolol; toloxatone; terbutaline; albuterol; phenobarbital; ranitidine; tiapride; phenol; chlormezanone; aspirin; metformin; ritodrine; codeine; sultopride; amisulpride; naltrexone; lisinopril; benzocaine; nizatidine; nalorphine; mephenesin; naloxone; sotalol; carteolol; procainamide; carbamazepine; bromazepam; nalbuphine; nadolol; procarbazine; dihydralazine; omeprazole; strychnine; acebutolol; glutethimide; chlorpropamide; glipizide; triazolam; prazosin; flunitrazepam; clonazepam; metoclopramide; melfalan; estazolam; tolbutamide; ephedrine; clonidine; pindolol; clobazam; minoxidil; disopyramide; nitrazepam; dextromethorphan; tofisopam; zopiclone; debrisoquine; sulindac; alprazolam; cycloguanil; lorazepam; methaqualone; ketamine; piroxicam; metoprolol; nifedipine; quinine; mephentermine; prilocaine; pentazocine; oxazepam; tiaprofenic acid; quinidine; celiprolol; ajmaline; yohimbine; lidocaine; secobarbital; viloxazine; mepivacaine; meperidine; doxylamine; labetalol; temazepam; amodiaquine; benperidol; droperidol; hydroxychloroquine; zolpidem; ketoprofen; alminoprofen; cicletanine; moclobemide; chloroquine; cocaine; timolol; nomifensine; ticlopidine; acenocoumarol; vandesine; mexiletine; dipyridamole; trazodone; pipamperone; pyrimethamine; benazepril; vincristine; metapramine; chlordiazepoxide; oxprenolol; warfarin; clorazepate; flecainide; phencyclidine; thiopental; fenfluramine; metipranolol; triprolidine; naproxen; buprenorphine; verapamil; buspirone; tianeptine; midazolam; bupivacaine; carbinoxamine; loprazolam; cetirizine; chlorpheniramine; moperone; cibenzoline; medifoxamine; astemizole; vinblastine; nicardipine; bisoprolol; diltiazem; glibornuride; reserpine; aconitine; nitrendipine; diazepam; mianserin; ramipril; haloperidol; tetracaine; alprenolol; aceprometazine; glibenclamide; chlorophenacinone; doxepin; nimodipine; diphenhydramine; cyclizine; histapyrrodine; phenylbutazone; demexiptiline; clozapine; proguanil; trifluoperidol; medazepam; cyamemazine; bumadizone; suriclone; propranolol; acepromazine; dothiepin; dextromoramide; fenopropfen; dextropropoxyphene; loxapine; betaxolol; propafenone; promethazine; thioproperazine; methadone; amoxapine; quinupramine; opipramol; cyproheptadine; brompheniramine; mefenidramine; protriptyline; flurbiprofen; tetrazepam; zorubicin; prazepam; alimemazine; loperamide; imipramine; desipramine; levomepromazine; hydroxyzine; niflumic acid; penbutolol; fluvoxamine; pimozide; daunorubicin; indomethacin; maprotiline; tropatenine; etodolac; fluoxetine; amitriptyline; nortriptyline; tiocloamarol; diclofenac; mefloquine; trimipramine; chlorambucil; lidoflazine; ibuprofen; floctafenine; alpidem; loratadine; chlorpromazine; clomipramine; carpi-pramine; thioridazine; fentiazac; clemastine; mefenamic acid; fluphenazine; prochlorperazine; penfluridol; bepridil; terfenadine; trifluoperazine

REFERENCE

Tracqui, A.; Kintz, P.; Mangin, P. Systematic toxicological analysis using HPLC/DAD, *J. Forensic Sci.*, **1995**, *40*, 254–262.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 × 4.6 Zorbax RX

Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.

Column temperature: 30

Flow rate: 2

Detector: UV 210

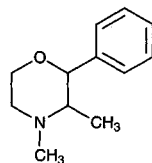
OTHER SUBSTANCES

Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitrityline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepam, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlor-diazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fencamfamine, fenpropofen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiaicol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, iminostilbene, imipramine, indomethacin, isocarboxystyryl, isocarboxazid, isoniazid, isoproterenol, isoxsuprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephénytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, methapyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyl dopa, methyl dopamine, methylphenidate, methylprednisolone, methyltestosterone, methyprylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, oxymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phendimetrazine, phenelzine, pheniramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrrolamine, pyrrhyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfamazepine, sulfamethazine, sulfamethoxazole, sulfanilamide, sulfapyridine, sulfasoxazole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranlycypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripeleminamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE

- Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233-242.

Phendimetrazine



Molecular formula: $C_{12}H_{17}NO$

Molecular weight: 191.27

CAS Registry No.: 634-03-7, 50-58-8 (tartrate)

Merck Index: 7365

Lednicer No.: 1 260

SAMPLE

Matrix: solutions

Sample preparation: Dissolve in MeOH at a concentration of 1 mg/mL, inject a 20 μ L aliquot.

HPLC VARIABLES

Column: 250 \times 5 Spherisorb S5W

Mobile phase: MeOH:buffer 90:10 (Buffer was 94 mL 35% ammonia and 21.5 mL 70% nitric acid in 884 mL water, adjust the pH to 10.1 with ammonia.)

Flow rate: 2

Injection volume: 20

Detector: UV 254

CHROMATOGRAM

Retention time: 1.74

OTHER SUBSTANCES

Simultaneous: hydroxypethidine, normethadone, meperidine, dipipanone, diamorphine, pentazocine, acetylcodeine, monoacetylmorphine, thebacon, oxycodone, thebaine, norlevorphanol, methadone, benzylmorphine, ethylmorphine, morphine-N-oxide, codeine, codeine-N-oxide, morphine, ethoheptazine, morphine-3-glucuronide, pholcodeine, norpethidine, hydrocodone, dihydrocodeine, dihydromorphine, levorphanol, norcodeine, normorphine, pemoline, benzphetamine, diethylpropion, epinephrine, pipradol, phenylpropanolamine, fencamfamin, chlorphentermine, norpseudoephedrine, phentermine, fenfluramine, methylenedioxymphetamine, amphetamine, normetanephrine, 4-hydroxyamphetamine, bromo-STP, STP, prolintane, 2-phenethylamine, tyramine, trimethoxyamphetamine, phenylephrine, pseudoephedrine, ephedrine, methylephedrine, dimethylamphetamine, methamphetamine, mescaline, mephentermine, buprenorphine, dextromoramide, phenoperidine, fentanyl, etorphine, piritramide, noscapine, papaverine

Noninterfering: dopamine, levodopa, methyl dopa, methyl dopate, norepinephrine

Interfering: mazindol, tranilcypromine, caffeine, fenethyline, methylphenidate, phenelzine, naloxone, dextropropoxyphene, nalorphine, phenazocine, norpipanone, levallorphan

REFERENCE

Law,B.; Gill,R.; Moffat,A.C. High-performance liquid chromatography retention data for 84 basic drugs of forensic interest on a silica column using an aqueous methanol eluent, *J.Chromatogr.*, **1984**, 301, 165-172.

SAMPLE

Matrix: solutions

Sample preparation: Prepare a 10 μ g/mL solution in MeOH, inject a 20 μ L aliquot.

HPLC VARIABLES

Column: 125 \times 4.9 Spherisorb S5W silica

Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7

Flow rate: 2

Injection volume: 20

Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM

Retention time: 1.6

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzocamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazepine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipiprone, diprenorphine, dipyrindamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscyne, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserine, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamlamine, meclorphenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylethylamphetamine, methysergide, metoclopramide, metopimazine, metoprolol, mianserine, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazoline, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, pimindine, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, propeptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranylecypromine, trazodone, trifluoperazine, trifluoperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripelethamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane, I.; McKinnon, A.; Flanagan, R. J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J. Chromatogr.*, **1985**, 323, 191–225.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 × 4.6 Zorbax RX

Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.

Column temperature: 30

Flow rate: 2

Detector: UV 210

OTHER SUBSTANCES

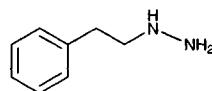
Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitriptyline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepam, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlor-diazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine,

chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fen-camfamine, fenpropofen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiaicol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, imino-stilbene, imipramine, indomethacin, isocarboxtyril, isocarboxazid, isoniazid, isoproterenol, isox-suprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, mepredine, mephentermine, mephenytoin, mephesis, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, meth-apyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyl dopa, methyl dopamine, methylphenidate, methylprednisolone, methyl-testosterone, methypyrrol, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, ox-ymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phenazopyridine, phenelzine, phenira-mine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenyl-ephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrila-mine, pyridylidone, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfa-merazine, sulfamethazine, sulfamethoxazole, sulfanilamide, sulfapyridine, sulfasoxazole, sulin-dac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, the-obromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranlycy-promine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripeleminamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE

Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233–242.

Phenelzine



Molecular formula: C₈H₁₂N₂

Molecular weight: 136.20

CAS Registry No.: 51-71-8, 156-51-4 (sulfate)

Merck Index: 7366

Lednicer No.: 1 74

SAMPLE

Matrix: blood

Sample preparation: 2 mL Plasma + 400 µL 10% acetic acid + 7 mL diethyl ether:dichloro-methane 2:1, shake, centrifuge at 2059 g for 10 min. Remove the aqueous layer and add it to 600 µL 10% acetic acid, add 300 µL 0.1% salicylaldehyde in EtOH, heat at 60° for 30 min, cool to room temperature, add 1 mL 1 M K₂PO₄ (sic), extract with 7 mL diethyl ether, centrifuge at 2059 g for 10 min, repeat extraction. Combine the organic layers and evaporate them to dryness under a stream of nitrogen at 40°, reconstitute the residue in 200 µL buffer, inject a 20 µL aliquot. (Buffer was 5 mM heptanesulfonic acid in MeCN:water:triethylamine 70:30:0.4.)